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## Long-term operation of a solid oxide cell stack for co-electrolysis of steam and CO<sub>2</sub>

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### Abstract

High temperature electrolysis based on solid oxide electrolysis cells (SOECs) is a promising technology for production of synthetic fuels. The SOEC units can be used for co-electrolysis of steam and CO<sub>2</sub> to produce synthesis gas (syngas, CO+H<sub>2</sub>), which can be further processed to a variety of synthetic fuels such as methane, methanol or DME. Previously we have reported electrolysis operation of solid oxide cell stacks for periods up to about 1000 hours. In this work, operation of a Haldor Topsoe 8-cell stack (stack design of 2014) in co-electrolysis mode for 6000 hours is reported. The stack consists of Ni/YSZ electrode supported SOEC cells with a footprint of 12X12 cm<sup>2</sup>. The co-electrolysis operation was carried out by supplying a mixture of 45 % CO<sub>2</sub> + 45 % H<sub>2</sub>O + 10 % H<sub>2</sub> to the stack operating with a fixed conversion of 39 % for steam and CO<sub>2</sub>. The stack was operated at different conditions. Initial operation at 700 °C and -0.25 A/cm<sup>2</sup> lasted for only 120 hours due to severe degradation of the bottom cell. Regaining the stack performance was realized by increasing the operation temperature to 750 °C. After reactivation, the stack showed negligible degradation at 750 °C and -0.25 A/cm<sup>2</sup> and about 1.4 %/1000 h performance degradation at 750 °C and -0.5 A/cm<sup>2</sup>. This study demonstrates feasibility of long-term co-electrolysis operation via SOEC stacks and of careful temperature variation as a tool to regain the stack performance.

## Introduction

High temperature electrolysis based on solid oxide electrolysis cells (SOECs) is a very promising technology for energy storage and production of synthetic fuels [1]. SOEC units can be used to convert excess electricity to energy carriers such as hydrogen or synthesis gas (syngas,  $H_2+CO$ ) via electrolysis of steam or co-electrolysis of steam and  $CO_2$ , respectively. The syngas could be further processed to methane which could be stored and distributed via the natural gas grid, or to synthetic fuels (synfuels) through the Fischer-Tropsch (F-T) process to be used in the transportation sector [2-3]. By combining SOEC with fuel production, electrical energy produced from renewable energy sources (wind, solar etc.) can be stored in chemical fuels and used in the transportation sector or be converted back into electricity at peak demand [4]. It is also worth noting that unlike conventional low temperature electrolysis (alkaline and PEM), SOEC is the only electrolysis technology allowing for co-electrolysis of steam and  $CO_2$  directly within the stack. The SOEC technology therefore offers a unique possibility for grid regulation in a future energy system with high amounts of fluctuating renewables.

An increased focus on developing the SOEC technology for hydrogen or syngas production has emerged in recent years, with different levels of R&D work ranging from materials level, to SOEC stacks, and further to SOEC system & plant design and simulation. For SOEC to become a competitive technology, durability is one of the crucial issues. As a core component of SOEC systems, the lifetime and durability of SOEC stacks are essential properties, which have not yet been demonstrated at a sufficiently high level. It is hence very important to acquire more experiences in the field. Performance and durability of SOEC stacks for electrolysis of steam or co-electrolysis of steam and  $CO_2$  have been investigated by a number of groups, with operating temperatures between 650 °C and 850 °C and current density up to  $-0.8\text{ A/cm}^2$  [5-11]. Most of the reported stack tests have been on steam electrolysis, with the longest one reported by Corre and Brisse, conducted on a Haldor Topsoe 25-cell stack [11]. The stack was tested at 750 °C (average stack temperature) and an electrolysis current density between 0.57 and  $0.72\text{ A/cm}^2$  for up to about 9000 h, with a degradation of 2 %/1000 h. We have also previously reported a number of stack tests using stacks from Haldor Topsoe [9-10]. The most recent test was conducted at 800 °C and  $-0.75\text{ A/cm}^2$  for co-electrolysis of steam and  $CO_2$ , with no notable degradation of the stack after 1000 h. Further reducing the stack operating temperature may bring several advantages, such as minimization of interconnect (IC) corrosion and therefore possibility of using inexpensive metallic interconnects, and minimization of reactions between different stack components. The aim of the current study was hence to explore operation limits of Haldor Topsoe's stacks at lower temperatures (700-800 °C) and examine the stack durability over a longer timeframe.

## 1. Experimental

### 1.1. Stack information

The stack used in the current study was produced by Haldor Topsoe. It is an 8-cell stack with a stack design of 2014. Planar type Ni/yttria stabilized zirconia (YSZ) electrode supported cells from same production batch were employed. The cells have a Ni/YSZ support, a Ni/YSZ active fuel electrode, an YSZ electrolyte, and a strontium and cobalt co-doped lanthanum ferrite (LSCF) / gadolinia doped ceria (CGO) oxygen electrode with a CGO barrier layer at the interface between the electrolyte and the oxygen electrode. Each cell has an active area of approximately  $87.7\text{ cm}^2$ . Further details about the cells can be

found elsewhere [12-13]. The cells were connected by interconnects (IC) made of Crofer22APU with a protective coating on both sides. The stack consists of eight serial repeating units (SRUs), numbered SRU01-08 with SRU01 located at the bottom of the stack. Voltage probes in contact with the IC plates were used to monitor the voltage over each SRU, which includes contributions from the cell, the IC, and the contact between them. In addition, the voltages between the stack end-plates (top- or bottom-plate) and the top or bottom SRU were monitored as well. Figure 1 presents a photo of the stack mounted in a furnace of an electrolysis stack test rig at DTU Energy. The stack temperature was monitored using eight thermocouples, with four inserted into the top-plate and the other four into the bottom plate. The locations are further illustrated in Figure 1.

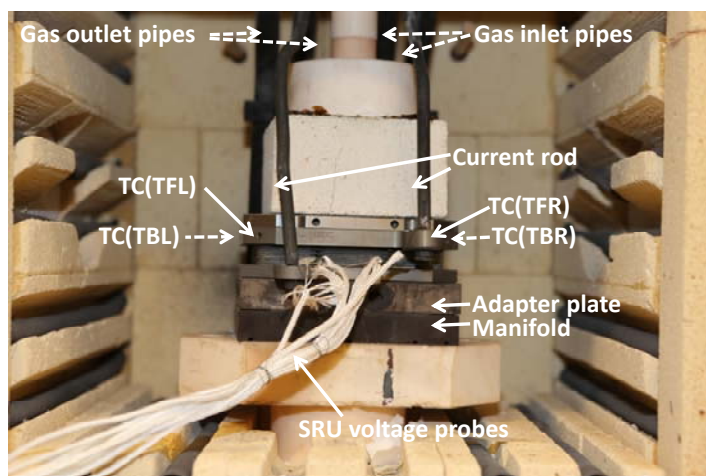


Figure 1: Photo of the 8-cell stack from Haldor Topsoe mounted in a furnace of an electrolysis stack test rig at DTU Energy. For monitoring the stack temperature, eight temperature probes (thermocouples) were inserted into the stack end-plates. The locations for the four inserted into the top-plate were indicated in the photo. The thermocouples were named using a 3-letter code: the first letter for top (T) or bottom (B) plate, the second letter for front-side (F, i.e. close to the current rod) or back-side of the stack, and the third letter for left-hand side (L) or right-hand (R) side of the stack.

## 1.2. Performance characterization and long-term durability testing

The stack was reduced at 800 °C, by supplying a mixture of H<sub>2</sub> and N<sub>2</sub> to the Ni/YSZ electrode compartment. Hereafter the stack performance was characterized by performing DC polarization (*iV*) curves at 800, 750, and 700 °C, with either H<sub>2</sub>O/H<sub>2</sub> (50/50) or CO<sub>2</sub>/H<sub>2</sub>O/H<sub>2</sub> (45/45/10) fed to the Ni/YSZ electrode compartment and pure oxygen to the LSCF/CGO electrode compartment. The detailed conditions are listed in Table 1. The following gasses from Air Liquide were used in the study: pure oxygen, industrial grade, O<sub>2</sub> ≥ 99.5%; hydrogen, N30, H<sub>2</sub> ≥ 99.9%; CO<sub>2</sub> ≥ 99.7%. Steam was produced by reacting oxygen with hydrogen. As in our previous studies [10], the gasses to the Ni/YSZ electrode were cleaned before entering the stack. The DC characterizations were conducted in both electrolysis cell mode (EC mode) and fuel cell mode (FC mode) for H<sub>2</sub>O/H<sub>2</sub> mixture, and in EC mode only for CO<sub>2</sub>/H<sub>2</sub>O/H<sub>2</sub> mixture. The area specific resistance (ASR) of the individual SRUs was calculated from the *iV* curves as the chord from open circuit voltage (OCV) to the voltage over the SRU measured at a current density of -0.15 A/cm<sup>2</sup> (EC mode) or +0.15 A/cm<sup>2</sup> (FC mode).

Table 1: List of conditions employed in stack performance characterization (*i*V measurements) and long-term durability testing.

Measurement type	Temp., °C	Gasses to Ni/YSZ		Oxygen to LSCF/CGO	Current density, A/cm <sup>2</sup>	Duration, hour
		Composition	Total flow, sccm/cm <sup>2</sup>	Total flow, sccm/cm <sup>2</sup>		
<i>i</i> V1	800	H <sub>2</sub> O/H <sub>2</sub> (50/50)	12.44	5.70	-	
<i>i</i> V2	800	CO <sub>2</sub> /H <sub>2</sub> O/H <sub>2</sub> (45/45/10)	6.21	2.85	-	
<i>i</i> V3	750	CO <sub>2</sub> /H <sub>2</sub> O/H <sub>2</sub> (45/45/10)	6.21	2.85	-	
<i>i</i> V4	700	CO <sub>2</sub> /H <sub>2</sub> O/H <sub>2</sub> (45/45/10)	6.21	2.85	-	
Durability1	700	CO <sub>2</sub> /H <sub>2</sub> O/H <sub>2</sub> (45/45/10)	4.97	2.85	-0.25	120
Durability2	750	CO <sub>2</sub> /H <sub>2</sub> O/H <sub>2</sub> (45/45/10)	4.97	2.85	-0.25	2200
<i>i</i> V5	750	CO <sub>2</sub> /H <sub>2</sub> O/H <sub>2</sub> (45/45/10)	4.97	2.85	-	
<i>i</i> V6	750	CO <sub>2</sub> /H <sub>2</sub> O/H <sub>2</sub> (45/45/10)	9.59	2.85	-	
Durability3	750	CO <sub>2</sub> /H <sub>2</sub> O/H <sub>2</sub> (45/45/10)	9.59	2.85	-0.5	3700

The durability of the stack during co-electrolysis of steam and CO<sub>2</sub> was investigated first at 700 °C and -0.25 A/cm<sup>2</sup>, but only for 120 hours (Durability1 in Table 1). The bottom SRU (SRU01) showed severe degradation. The stack temperature was then increased to 750 °C, which enabled a regaining of the stack performance. The stack was operated at this condition (750 °C and -0.25 A/cm<sup>2</sup>, Durability2 in Table 1) for approximately 2200 hours, showing negligible degradation. Afterwards, the stack was operated at 750 °C and -0.5 A/cm<sup>2</sup> for another 3700 h (Durability3 in Table 1). Between Durability2 and Durability3, two additional DC characterizations were conducted as ramping down and up the current. The test was terminated accidentally after 6000 h due to an instrument failure, which unfortunately resulted in failure of the stack. During the three durability test periods, the gas flow to the Ni/YSZ electrode compartment was adjusted to ensure same conversion, while the oxygen flow to LSCF/CGO was kept constant. Based on the Faraday's law, the conversion of steam and CO<sub>2</sub> was calculated to be 39 %.

## 2. Results and discussion

### 2.1. Initial performance

After reduction, the stack performance was characterized first at 800 °C with a mixture of H<sub>2</sub>O/H<sub>2</sub> (50/50) supplied to the Ni/YSZ electrode and oxygen supplied to the LSCF/CGO electrode. The measured OCV for the eight SRUs was within 0.973-0.974 V, very close to the theoretical EMF (0.977 V), indicating good sealing of the stack. The DC characterization was conducted in both EC and FC modes. The average ASR value calculated from *i*V curves was 0.31 Ω cm<sup>2</sup> in FC mode and 0.37 Ω cm<sup>2</sup> in EC mode, similar to what was reported in our previous study [10]. The stack performance for co-electrolysis of steam and CO<sub>2</sub> was then characterized at 800, 750, and 700 °C, by performing DC characterizations in EC mode only with a mixture of CO<sub>2</sub>/H<sub>2</sub>O/H<sub>2</sub> (45/45/10) supplied to the Ni/YSZ electrode and oxygen supplied to the LSCF/CGO electrode. Figure 2 presents the *i*V curves of the eight SRUs and evolution of the stack temperature for the measurement at 800 °C. The calculated ASR values for all the three temperatures are presented in Figure

3. At 800 °C, the measured OCV of the individual SRUs was within 0.877-0.880 V, slightly higher than the theoretical EMF (0.875 V). This difference could be due to small deviations of temperature and the gas composition from the desired ones (800 °C and 45%CO<sub>2</sub>+45%H<sub>2</sub>O+10%H<sub>2</sub>). Unlike the measured OCV, the ASR of the individual SRUs shows a relatively large scatter. SRU01, 05, and 07 showed much higher ASR values, while SRU04 and SRU08 showed lower, and the same trend was maintained for all the three temperatures. As described in Section 1 Experimental and in Figure 1, the stack temperature was monitored using eight thermocouples inserted into different locations in the bottom and top plates. Inside the furnace, the stack was heated from the left, right and bottom sides. At OCV, the stack has the hottest part at TBL (top-back-left) and TBR (top-back-right) and the coldest part at BFL (bottom-front-left). When the electrolysis current was applied, the stack temperatures first decreased due to endothermal electrolysis reactions, then increased due to Joule heating. The minimum was at around -0.2-0.3 A/cm<sup>2</sup>, corresponding to an average SRU voltage of 1.0-1.1 V. At -0.5 A/cm<sup>2</sup>, the corner at TFR (top-front-right) became the hottest. TFR has also the largest temperature increase when increasing the current density from zero to -0.5 A/cm<sup>2</sup>. The top plate was in general 1-2 °C hotter than the bottom plate.

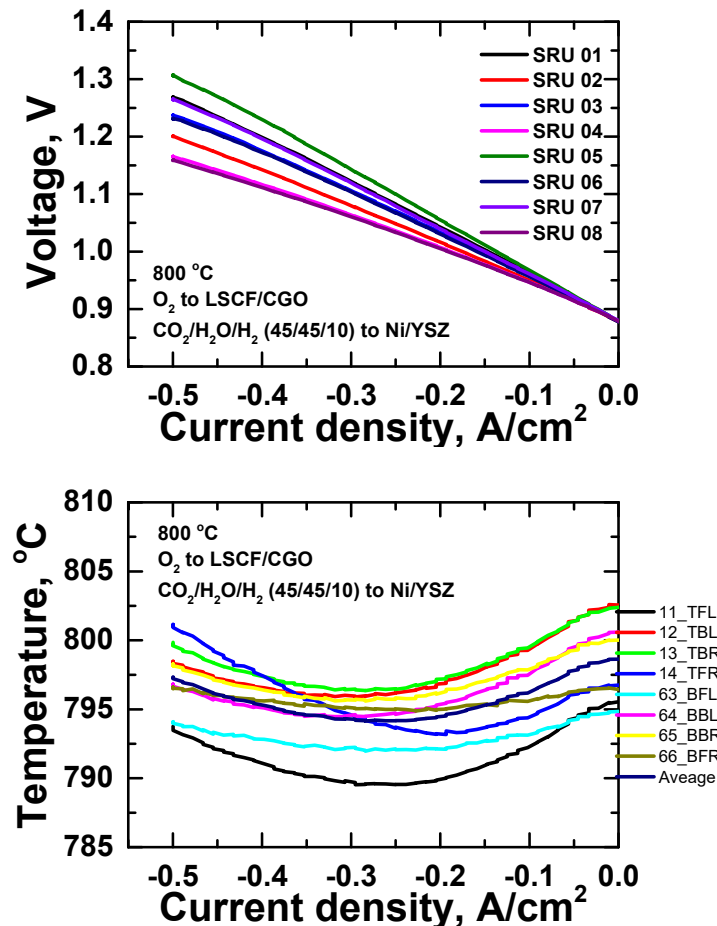


Figure 2: *iV* curves of the eight SRUs in the stack and temperature measurements for *iV*2 at 800 °C with CO<sub>2</sub>/H<sub>2</sub>O/H<sub>2</sub> (45/45/10, at a total flow rate of 6.21 sccm/cm<sup>2</sup>) supplied to the Ni/YSZ electrode and oxygen (at a total flow rate of 2.85 sccm/cm<sup>2</sup>) to LSCF/CGO.

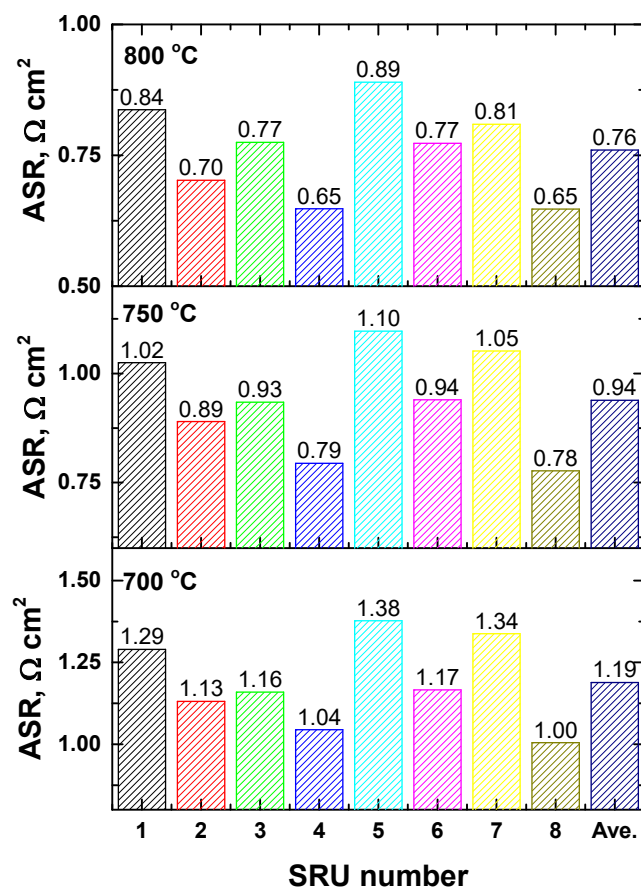


Figure 3: ASR values of the eight SRUs plus the average evaluated from the  $i/V$  curves ( $i/V_{4/3/2}$ ) measured at 700, 750, and 800 °C with  $\text{CO}_2/\text{H}_2\text{O}/\text{H}_2$  (45/45/10, at a total flow rate of 6.21 sccm/cm<sup>2</sup>) supplied to the Ni/YSZ electrode and oxygen (at a total flow rate of 2.85 sccm/cm<sup>2</sup>) to LSCF/CGO.

Figure 4 plots the average voltage of the eight SRUs and – based on that – the stack power density at different temperatures. For calculating the stack power density, the contribution from the end-plates was excluded. According to Sun et al. [14], the thermo-neutral voltage for co-electrolysis of steam and  $\text{CO}_2$  is around 1.367 V. At -0.5 A/cm<sup>2</sup>, the average SRU voltage was measured as 1.40, 1.31, 1.23 V at 700, 750, and 800 °C, respectively, close to or below the thermo-neutral voltage. The stack power density (per cm<sup>2</sup> cell area) was calculated as 0.70, 0.66, 0.61 W/cm<sup>2</sup> accordingly. By decreasing stack operating temperature with 50 °C and keeping the same current density (i.e. same syngas production rate), an increase of 5-10 % in stack power consumption is expected.

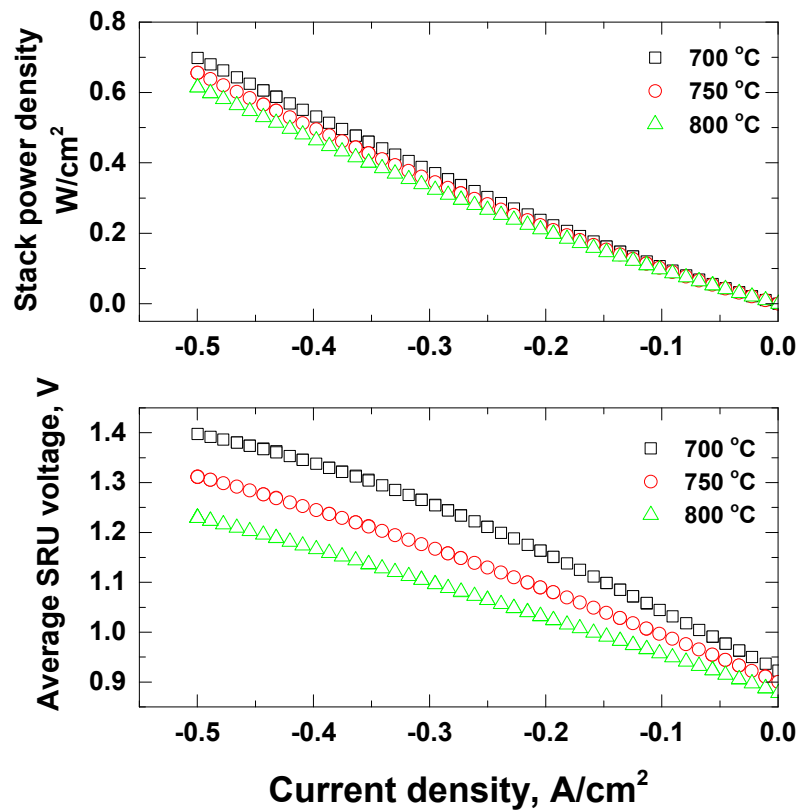


Figure 4: Average SRU voltage and stack power density calculated from the  $i/V$  curves ( $iV_{4/3/2}$ ) measured at 700, 750, and 800 °C with  $\text{CO}_2/\text{H}_2\text{O}/\text{H}_2$  (45/45/10, with a total flow rate of 6.21 sccm/ $\text{cm}^2$ ) supplied to the Ni/YSZ electrode and oxygen (with a total flow rate of 2.85 sccm/ $\text{cm}^2$ ) to LSCF/CGO.

## 2.2. Durability test

The durability of the stack for co-electrolysis of steam and  $\text{CO}_2$  was tested at 3 different conditions: 700 °C and  $-0.25 \text{ A/cm}^2$  (Durability1), 750 °C and  $-0.25 \text{ A/cm}^2$  (Durability2), and 750 °C and  $-0.5 \text{ A/cm}^2$  (Durability3). Figure 5 presents an overview of the stack voltage and the average stack temperature over the entire 6000 h testing period. The voltages of the individual SRUs are plotted in Figures 6 and 8. Durability1 lasted for only 120 hours. The bottom SRU (SRU01) showed fast degradation, especially in the last 10 hours of Durability1 where its voltage increased from 1.3 V to above 1.6 V. The other SRUs did not seem to be affected. The cause of fast degradation associated with SRU01 is unclear. It is speculated that it could be due to poor contacting between Cell01 and the neighboring ICs or between SRU01 and the neighboring current collector, resulting in uneven current distribution. The test at 700 °C and  $-0.25 \text{ A/cm}^2$  was then terminated. Instead of going back to OCV, the current ( $-0.25 \text{ A/cm}^2$ ) was maintained and the stack temperature was increased to 750 °C. The 50 °C increase in stack operation temperature made a sufficient improvement in stack performance (mostly likely due to improved contacting) to be able to continue the testing. The three SRUs, SRU01/05/07, which had the highest ASR among the eight SRUs during initial DC characterizations (Figure 3) and which had much higher voltage than the rest at the beginning of Durability2, all showed large activation between 120 h and 500 h. The activation of SRU01 lasted much longer, up to around 1200 h. The



rest of the SRUs showed much smaller changes in voltage over time. The average stack temperature changed only 1-2 °C over the 2200 h testing of Durability2.

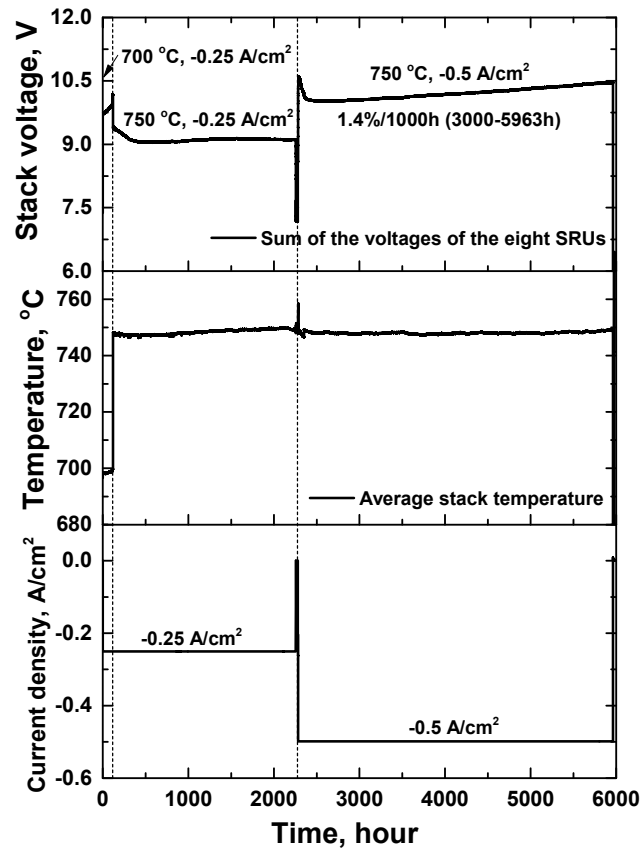


Figure 5: Evolution of the stack voltage and temperature during the 6000 h durability test period.

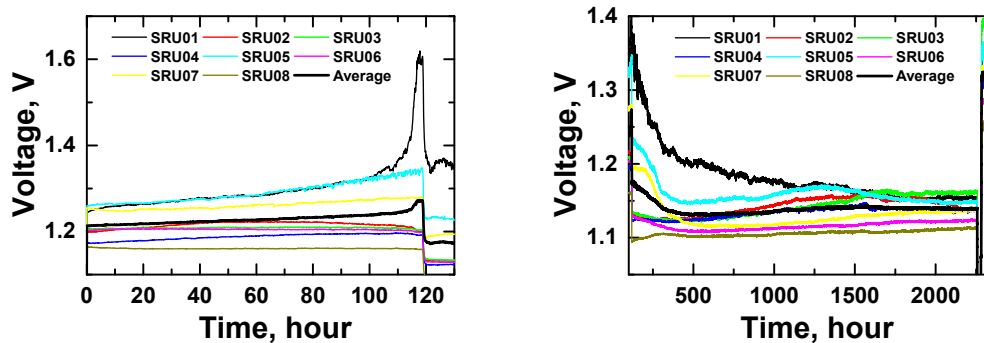


Figure 6: Evolution of the SRU voltages for Durability1 (700 °C and -0.25 A/cm²) and Durability2 (750 °C and -0.25 A/cm²).

At the end of Durability2, the stack was brought to OCV and shortly after to -0.5 A/cm². Two DC characterizations (*i*V5/6) were conducted during stepping down and stepping up the current. The OCVs of the eight SRUs were measured to be 0.900±0.002 V, very close to the theoretical EMF (0.897 V). The measured OCV values are most identical to those obtained in *i*V3, indicating that good sealing of the stack was maintained after 2300 h

testing. The evaluated ASR values from  $iV5$  were plotted in Figure 7, in comparison with those from  $iV3$ . As discussed earlier, the eight SRUs showed a large scatter in ASR values before durability testing ( $iV3$ :  $0.94 \pm 0.16 \Omega \text{ cm}^2$ ). After 2300 h durability testing, the SRUs showed an ASR of  $0.98 \pm 0.10 \Omega \text{ cm}^2$  ( $iV5$ ), with a much smaller scatter. The ASR values presented in this work were not corrected with fuel utilization. By taking into account of different flow rates in  $iV3/5$ , it can be concluded that the average performance of the SRUs at 2300 h was almost identical to that at 0 h. Those with better initial performance (SRU02/03/04/08) degraded, while SRU01/05/07 first degraded during Durability1 and afterwards activated during Durability2.

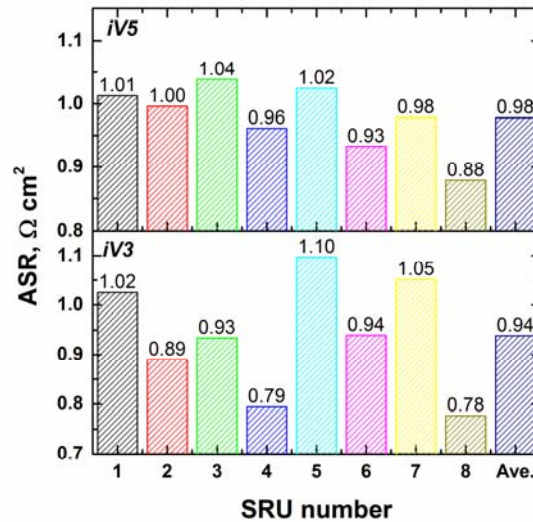


Figure 7: ASR values of the eight SRUs evaluated from the  $iV$  curves ( $iV3/5$ ) measured at 750 °C with  $\text{CO}_2/\text{H}_2\text{O}/\text{H}_2$  (45/45/10) supplied to the Ni/YSZ electrode and oxygen to LSCF/CGO. The conditions employed in the  $iV$  measurements can be found in Table 1.

After stepping the current up to  $-0.5 \text{ A/cm}^2$ , the SRUs showed a voltage within 1.2-1.4 V. The average stack temperature was 746 °C. The furnace temperature was then increased by 5 °C, resulting in an average stack temperature of 750 °C. This 5 °C temperature increase resulted in a significant improvement in stack performance, bringing down the average SRU voltage from 1.35 V to 1.25 V. After being at 750 °C and  $-0.5 \text{ A/cm}^2$  for 300-400 h, all the SRUs started degrading rather linearly, with degradation rates ranging from 10 mV/1000 h to 30 mV/1000 h. The average degradation rate for the period of 3000-5963 h was 17 mV or 1.4% per 1000 h.

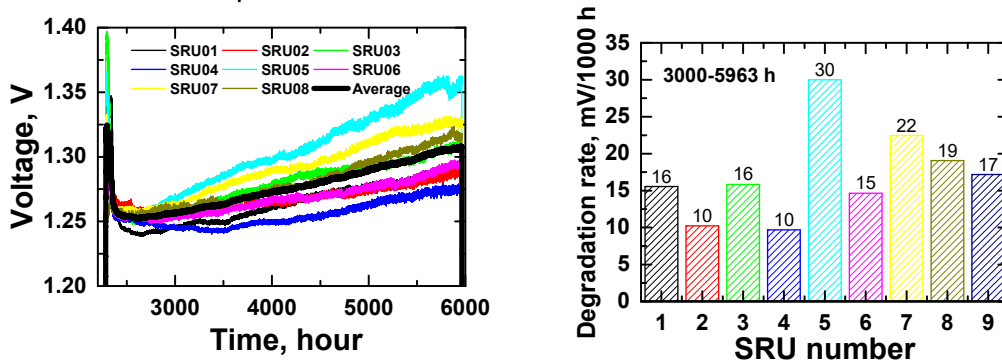


Figure 8: Evolution of the SRU voltages and calculated voltage degradation rates during the period when the stack was tested at 750 °C and  $-0.5 \text{ A/cm}^2$ .

Figure 9 plots the end-plate resistance calculated from the measured voltage loss. The two end-plates had similar resistance initially ( $\sim 0.7 \Omega \text{ cm}^2$ ) and both remained stable during Durability1. However, the top-plate resistance increased sharply and reached close to  $4 \Omega \text{ cm}^2$  at the end of Durability2. The  $iV$  measurements ( $iV5/6$ ) between Durability2 and Durability3 helped reducing the top-plate resistance to  $\sim 2 \Omega \text{ cm}^2$ , which then remained stable over Durability 3. On the contrary, the bottom plate resistance remained rather stable or actually slightly decreased over time and ended up with  $0.4 \Omega \text{ cm}^2$  at 6000 h. It is most likely that the higher contact resistance at the top plate contributed (at least partially) to higher temperature measured there. Corre and Brisse [11] tested a Haldor Topsoe 25-cell stack with similar design (stack design of 2014) for steam electrolysis over 9000 h. In their study, the top-plate resistance remained almost unchanged while the bottom-plate resistance showed fast increase and after an accidental improvement remained two times higher than that of the top-plate. It seems that the contacting at the end-plates remains a critical issue for the 2014 stack design and that there is no consistent picture on which of the two end-plates has the best contacting.

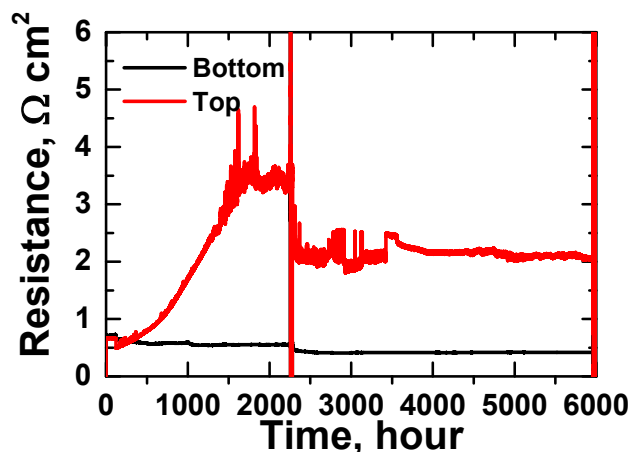


Figure 9: Evolution of the resistance across the end-plates during the 6000 h durability test period.

At 6000 h, the test was accidentally terminated due to an instrument failure, where pure oxygen was fed into the Ni/YSZ electrode compartment and hence destroyed the stack. However, the current test does demonstrate a sufficiently high reliability for the Haldor Topsoe stacks (stack design of 2014) for co-electrolysis of steam and  $\text{CO}_2$ . In this work, a degradation rate of 17 mV or 1.4% per 1000 h was demonstrated for co-electrolysis at 750 °C and  $-0.5 \text{ A/cm}^2$ . Assuming an end-of-life SRU voltage of 1.5 V, a lifetime of >15000 hours is probably achievable. A new stack test under similar conditions is currently being started, targeting a demonstration of >1 year co-electrolysis operation. The current study indicates that for the 2014 stack design,  $-0.5 \text{ A/cm}^2$  is the upper limit with regard to current density for co-electrolysis operation at 750 °C, if a lifetime of >2 years is required.

### 2.3. Stack power consumption

Figure 10 plots the stack power consumption, in terms of kWh electricity per  $\text{Nm}^3$  syngas production, calculated from either DC characterizations ( $iV$  measurements) or durability test data. To keep simplicity, here we take into account only the power consumption directly by the SOEC stack. It is obviously desirable and advantageous to operate SOECs at high current density in order to decrease investment cost. However, with increasing

current density, the kWh electricity consumption will increase. As shown in Figure 10, at 750 °C, the electricity consumption increased from 2.7 kWh/Nm<sup>3</sup> at -0.25 A/cm<sup>2</sup> to 3.1 kWh/Nm<sup>3</sup> at -0.5 A/cm<sup>2</sup>. Besides, higher current density often means higher degradation and hence shorter lifetime, as reflected in Figure 10. A detailed life-time efficiency and cost analysis requires information such as SOEC stack performance and durability, SOEC system lay-out, investment cost, electricity price etc. In addition, additional power/heat input from the furnace or other balance-of-plants (BOP) components should be taken into account which is neglected here. This analysis is out of the scope for the current paper and will be presented elsewhere.

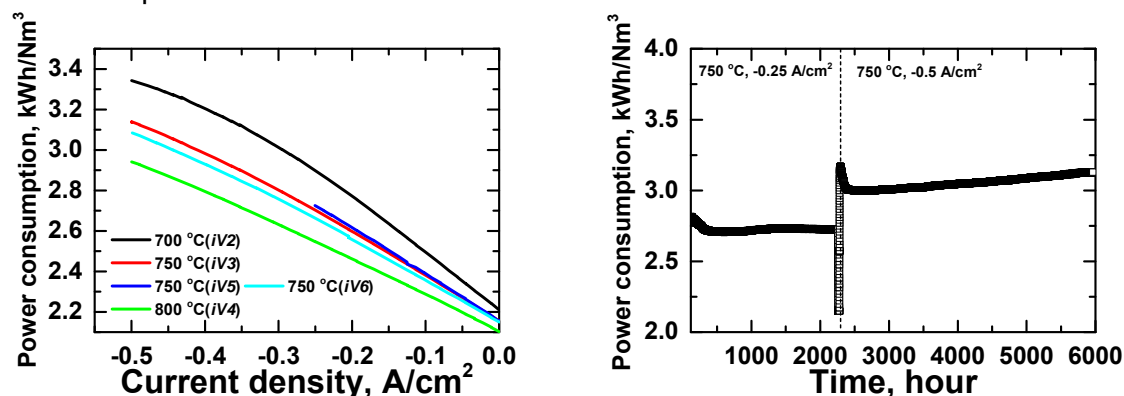


Figure 10: SOEC stack power consumption (kWh electricity consumption per Nm<sup>3</sup> syngas production) derived from either stack *iV* measurements (left) or durability test data for the period at 750 °C, -0.25 and -0.5 A/cm<sup>2</sup> (right).

### 3. Conclusion

In this work, co-electrolysis of steam and carbon dioxide was demonstrated in a Haldor Topsoe 8-cell stack with a stack design of 2014. The stack was operated under co-electrolysis operation for 6000 h in total. Three operating points were chosen for durability testing, namely 700 °C and -0.25 A/cm<sup>2</sup>, 750 °C and -0.25 A/cm<sup>2</sup>, and 750 °C and -0.5 A/cm<sup>2</sup>. Due to severe degradation of the bottom SRU (SRU01), testing at 700 °C and -0.25 A/cm<sup>2</sup> lasted for only 120 h. Increasing the operation temperature to 750 °C enabled a stack performance recovery (mostly likely due to improved contacting within the stack) and smooth electrolysis operation with almost no degradation (average) after 2200 h. When operated at 750 °C and -0.5 A/cm<sup>2</sup>, the stack showed an average degradation rate of 1.4 %/1000 h. The current work demonstrates feasibility of long-term co-electrolysis operation via SOEC stacks and of careful temperature variation to regain the stack performance.

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